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硕 士 学 位 论 文

电泳沉积羟基磷灰石涂层以及涂层表面修饰研究  
Investigation for Electrophoretic Deposition of Hydroxyapatite  
Coatings and the Surface Modification of Coatings

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## 摘 要

羟基磷灰石 ( $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ , HA) 是人骨等硬组织的主要无机成分, 具有良好的生物相容性和高的生物活性, 被广泛用作为生物医用植入材料。然而, HA陶瓷材料的力学性能较差, 限制了它在生物医学领域的应用。为此人们致力于发展各种HA涂层或复合材料。在医用金属上涂覆HA涂层是目前复合生物医学材料领域研究的热点之一。电泳沉积法可在复杂形貌的基体上获得均匀的涂层并且涂层的结构和成分可控。这些独特的优势使其在HA陶瓷涂层的制备中引起了广泛的兴趣。但是电泳沉积法制备HA陶瓷涂层普遍存在着结合强度不够高的问题。为了改善电泳沉积法制备的HA涂层的结合强度并进一步提高HA涂层的生物学性能, 本论文在实验室课题组已取得的成果的基础上, 对电泳沉积HA涂层的工艺以及电泳沉积过程中的基本问题进行研究, 并尝试对HA涂层进行表面修饰。主要研究内容如下:

一、以无水乙醇作为分散介质, 研究悬浮液特性参数的变化规律及其对电泳沉积 HA 涂层的影响。采用扫描电镜 (SEM) 观察涂层的表面形貌。结果表明, 悬浮液的特性参数随悬浮液 pH 的改变表现出一定的变化规律。悬浮液的平均粒径随 pH 的升高而增大; Zeta 电位随 pH 的升高表现出先增加后减小的趋势, 并在 pH 2.9 附近达到最大值; 电导率随 pH 的增大先减小后增大, 并在 pH 7.4 附近取得最小值; 悬浮液在酸性条件下 (pH 1.4~6.9) 表现出良好的稳定性, 在碱性条件下悬浮液稳定性较差。另外, 悬浮液的 Zeta 电位表现出一定的陈化作用, 即悬浮液的 Zeta 电位在前三天有一定的增幅, 之后基本保持恒定。悬浮液特性参数对电泳沉积过程具有重要影响。在恒压模式下, 电泳沉积速率随 pH (pH 1.4~6.9) 的增大表现出先增大后减小的变化趋势, 并在 pH 2.9 附近达到最大值。此外, 沉积速率还受陈化时间的影响, 即沉积速率在前三天表现出一定的增幅, 之后保持不变。针状羟基磷灰石颗粒在电泳过程中容易导致涂层产生裂纹。在 pH 2.9 附近下电泳沉积获得的涂层的裂纹较少。悬浮液在 pH 2.9 附近 (电导率为  $11 \sim 12 \mu\text{s}\cdot\text{cm}^{-1}$ ) 且陈化 1~2 d 是一个合适的电泳沉积条件。

二、研究涂层裂纹的产生并提出抑制涂层裂纹产生的方法。基于颗粒双电层

扭曲和变薄的沉积机理和涂层裂纹产生的实验现象,对涂层裂纹的产生进行了定性的描述。随着沉积层厚度的增加,双电层中离子的传导受阻,导致离子不能充分参与电极反应而被消耗。离子传导受阻使沉积层外层颗粒双电层减薄程度下降,溶剂化程度较高,颗粒间距增大。由此导致涂层随厚度的增加而遭受更大程度的涂层开裂。多次沉积的方法有利于抑制涂层裂纹的产生。随着溶剂的挥发,沉积层中颗粒表面电荷减少,双电层变薄,颗粒间距进一步缩小,减小了沉积层的电阻,有利于下次沉积的进行,从而抑制了涂层裂纹的产生。

三、采用电泳沉积-反应烧结法制备了HA/Al<sub>2</sub>O<sub>3</sub>复合涂层,并探讨复合涂层组成对涂层结构和性能的影响。采用划痕试验表征涂层的结合强度。结果表明,通过改变悬浮液中Al粉的含量,可实现HA/Al复合涂层化学组成的调控。反应烧结的应用有效地提高了HA/Al<sub>2</sub>O<sub>3</sub>复合涂层的烧结程度和致密程度。涂层的致密程度随涂层Al元素含量的增加而增加,因此通过改变悬浮液中Al粉的含量,可实现HA/Al<sub>2</sub>O<sub>3</sub>复合涂层结构的控制。HA/Al<sub>2</sub>O<sub>3</sub>复合涂层的结合强度随着涂层Al元素含量的增加表现出先增加后下降的趋势。悬浮液Al粉含量在10~20 wt%的范围内是一个适宜的电泳沉积工艺,在该条件下制备的HA/Al<sub>2</sub>O<sub>3</sub>复合涂层具有较高的结合强度。

四、采用仿生矿化法对电泳沉积制备的HA涂层进行表面修饰以期进一步提高涂层的生物学性能。首先,通过胶原的自组装在HA表面涂覆了一层胶原薄膜。接着,采用仿生矿化法对胶原涂覆层进行固定。胶原的涂覆和仿生矿化法的应用,实现了HA涂层表面有机/无机复合物的修饰。

**关键词:** 涂层 电泳沉积 羟基磷灰石 悬浮液 表面修饰

## Abstract

Hydroxyapatite ( $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ , HA), the major inorganic component of human bone and other hard tissues, has been used extensively for biomedical implant applications due to its good bioactivity and excellent biocompatibility. However, the poor mechanical properties of HA ceramic have limited its clinical applications. For this reason, a great deal of research has concentrated on the development of HA coatings and/or composites. Special attentions have been devoted to deposit HA coatings on the surface of biomedical metals in the field of biomedical materials. There is growing interest in the fabrication of HA coatings by the process of electrophoretic deposition (EPD), due to its ability to form uniform coatings on complex substrate and flexibility to control the structure and composition of coatings. The main problem encountered in the fabrication of HA coatings by EPD is the unsatisfactory bonding strength between the coatings and substrates. This work focuses on the further improvement of both the bonding strength and biological performance of HA coatings. Experiments were conducted to study the process of electrophoretic deposition of HA coatings as well as the related fundamentals in electrophoretic deposition, basing on the previous results gained in our lab. Moreover, an attempt was taken to modify the surface of HA coatings to enhance the biological performance of HA coatings. Researches involved in this work are summarized below.

Firstly, absolute ethanol was used as dispersing medium and the parameters related to the suspension property were examined in details. The influences of suspension properties on the EPD of HA coatings were studied and scanning electron microscope (SEM) was used to observe the surface morphology of HA coatings. Results show that the pH played a significant role in the properties of the suspension. The average size of particles increased as pH increased. Zeta potential firstly increased, gaining a maximum at pH 2.9 and then decreased as pH increased. The conductivity of suspension firstly decreased and then increased as pH increased with a minimum at pH 7.4. The suspension exhibited a good stability under the acidic condition (pH 1.4~6.9) and lost its stability under a basic condition. In addition, Zeta potential showed an aging effect, namely, gaining an increase in magnitude in the first three

days and then keeping nearly constant. The parameters related to the suspension property had a significant influence on the process of EPD. The deposition rate under a constant-voltage EPD increased firstly, gaining a maximum at pH 2.9, and then decreased as pH increased in the range of pH 1.4~6.9. In addition, the deposition rate was affected by aging time and showed an increase in the first three days. The coatings deposited from a suspension of needle-like HA were ready to be cracking. The crack was minimized at pH 2.9. The suspension with pH~2.9 (conductivity about 11~12  $\mu\text{s}\cdot\text{cm}^{-1}$ ) and an aging time of 1~2 d is suitable for EPD.

Secondly, the formation of cracks was studied and the method to control cracks was presented. Basing on the deposition mechanism of double layer distortion and thinning and the experimental observation of cracks, the formation of cracks was illustrated by a qualitative description. The transmission of ions in the double layer is hindered as the growing deposit, leading to an insufficient depletion of ion by electrode reaction. This reduces the thinning degree of double layer for particles in the outer layer of deposit, increasing both the salvation of particles and separation between two particles in contrast to that of inner layer of deposit. As a result, cracks increase as the thickness of coatings grows. Multi-deposition method can be used to control the cracks. As the vaporization of solvent, the surface charge of particles in the deposit is reduced, decreasing the thickness of double layer, diminishing the particle distance and as a result leading to a lower resistance of deposit which paves the way for the subsequent deposition and restrains the formation of cracks.

Thirdly, the combination of electrophoretic deposition and reaction bonding was applied to prepare HA/Al<sub>2</sub>O<sub>3</sub> composite coatings and the relationship between the composition and structure of composite coatings was studied. Scratch test was used to estimate the bonding strength. Results show that the chemical composition of HA/Al composite coatings can be adjusted by the variation in the Al powder content of suspensions. The sintering degree and density of HA/Al<sub>2</sub>O<sub>3</sub> composite coatings were significantly enhanced by the application of reaction bonding process. As the density of composite coatings increased with the increasing content of Al element, the structure of composite coatings can be controlled by the variation in the Al powder content of suspensions. The bonding strength of HA/Al<sub>2</sub>O<sub>3</sub> composite coatings firstly increased and then decreased as the content of Al element increased. The suspension with the content of Al powder in the range of 10~20 wt% is suitable for the

fabrication of HA/Al<sub>2</sub>O<sub>3</sub> composite coatings and such prepared coatings gain a relatively high bonding strength.

Finally, biomimetic mineralization was utilized to modify the surface of HA coatings prepared by electrophoretic deposition. Collagen was deposited onto the surface of HA coatings by the self-assembly of collagen. The collagen layer on the HA coatings was subsequently fixed by biomimetic mineralization. After this two step, the surface of HA coatings was modified by a layer of organic/inorganic composite.

**Keywords:** coatings; electrophoretic deposition; hydroxyapatite; suspension; surface modification

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## 第一章 绪论

### 1.1 生物医用材料的概念及分类

生物医用材料 (Biomedical materials) 又称生物材料 (Biomaterials), 是用来对生物体进行诊断、治疗和置换损坏的组织、器官或增进其功能的材料<sup>[1]</sup>。它是研究人工器官和医疗器械的基础, 已成为各国科学家竞相进行研究和开发的热点<sup>[2]</sup>。随着人口老龄化进程的加速和人类对健康的追求, 生物医用材料的需求将不断增长。生物医用材料的发展和进步无疑将进一步提高人们的健康水平和生活质量, 对社会产生深远的影响。

人类利用生物医用材料的历史可追溯到几千年前<sup>[2]</sup>。公元前约 3500 年古埃及人就利用棉花纤维、马鬃作缝合线缝合伤口。公元前 2500 年前中国、埃及的墓葬中就发现有假牙、假鼻、假耳。人类很早就用黄金来修复缺损的牙齿。但生物医用材料的迅猛发展是在第二次世界大战之后才开始的<sup>[3]</sup>。20 世纪 40 年代首次报道将塑料 (合成高分子) 植入人体的案例。当时关于高分子的所有研究都集中于聚甲基丙烯酸甲酯和尼龙这两种合成高分子材料上。随后, 生物医用材料迅速发展, 包含各种类型的材料。第二次世界大战之后的 20 年中, 人工髋关节 (金属医用生物材料)、肾透析仪 (纤维素) 和人造血管 (丝绸) 等相继成功问世。

根据生物医用材料的发展史及所用的材料, 可将其分为三代<sup>[3,4]</sup>。第一次世界大战以前所使用的可归为第一代生物医用材料。这一代生物医用材料主要是惰性的, 或者说不与人体发生反应, 代表材料有石膏、各种金属、橡胶以及棉花等。到 20 世纪 90 年代, 这一概念逐渐被第二代生物医用材料, 即生物活性材料所替代, 生物活性材料能与人体发生积极的相互作用, 促进组织的局部愈合。其代表材料有羟基磷灰石、磷酸三钙、聚羟基乙酸、聚甲基丙烯酸羟乙酯、胶原、多肽、纤维蛋白等。随着 21 世纪的到来, 出现了第三代生物医用材料。它是一类具有促进人体自身修复和再生的生物医学复合材料, 一般由具有生理活性的组元和控释载体的非活性组元构成。骨形态基因蛋白 (Bone morphogenetic protein, BMP) 材料是第三代生物医用材料中的代表。

生物医用材料种类繁多, 到目前为止, 被详细研究过的生物医用材料已经超过 1000 中, 在医学临床上广泛应用的也有几十种<sup>[4]</sup>。按材料组成和性质可分为

生物医用金属材料、生物医用高分子材料、生物医用陶瓷材料以及由它们组合而成的生物医用复合材料<sup>[5]</sup>。

### 1.1.1 生物医用金属材料

金属材料是人类最早利用的生物医用材料之一，具有高的机械强度、良好的韧性和抗弯曲疲劳强度、优异的加工性能等优势，是临床应用最广泛的承力植入材料<sup>[6]</sup>。该材料重要的应用包括骨折内固定板、螺钉、人工关节和牙根植入体等。目前临床上应用较多的金属材料有：不锈钢、钴基合金、钛及其合金等。

钛及其合金是目前最理想的生物医用金属植入材料，在人工髋关节、膝关节和肩关节等方面发挥着重要的作用<sup>[7]</sup>。在生物体环境中，316L 不锈钢和 Co-Cr 合金因腐蚀而释放出 Ni、Cr 和 Co 等元素<sup>[8]</sup>，并由此导致毒副作用。另外，316L 不锈钢和 Co-Cr 合金具有比人骨高的弹性模量，导致生物机械不相容<sup>[7]</sup>。与 316L 不锈钢和 Co-Cr 合金相比，钛及其合金集众多优异性能于一身，使其更加适合于植入材料的应用：具有高比强度，良好的耐蚀性，在生物体环境中完全惰性，优良的生物相容性以及弹性模量低（与人骨最为接近）等<sup>[9]</sup>。在美国、欧洲等发达国家，钛及其合金在矫形外科、硬组织修复等方面已经逐渐取代了不锈钢和钴基合金的统治地位。

尽管生物医用金属材料具有优良的力学性能，但它本质上属于生物惰性材料。作为骨植入材料时，生物医用金属材料不能与骨组织发生化学键性结合，容易松动而导致植入失败。生物医用金属材料存在的另一个主要问题是：由于生理腐蚀造成的金属离子向周围组织扩散及植入材料自身性质的蜕变可能导致毒副作用甚至植入失败<sup>[6]</sup>。利用表面改性技术提高金属材料的耐蚀性和生物活性是目前生物医用金属材料发展的主要方向<sup>[10, 11]</sup>。

### 1.1.2 生物医用陶瓷材料

生物医用陶瓷材料作为无机生物医学材料，没有毒副作用，与生物体组织有良好的生物相容性，越来越受到重视。根据陶瓷在生物体内的活性，可将其分成三类<sup>[12]</sup>：生物惰性陶瓷，生物可降解陶瓷，生物活性陶瓷。

生物惰性陶瓷具有稳定的化学性能和良好的生物相容性，在体内能耐氧化、耐腐蚀，不降解，与骨组织不能产生化学结合。另外，这类材料具有较高的机械



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